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NAVY EXPERIMENTAL DIVING UNIT

REPORT NO. 01-98

LOT VARIABILITY OF SOFNOLIME 408 CARBON DIOXIDE ABSORBENT WHEN TESTED IN THE COLD

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NEDU performed quality assurance tests on 18 lots of Sofnolime 408, a large grain sodalime made by Molecular Products. CO ₂ absorption activity using the standard NATO tube test was performed at initial absorbent bed temperatures of approximately 32° F. Moisture analyses and sieve tests were also performed on each sample. Absorption test breakthrough in the chilled bed occurred at an average of 7 min, compared to the usual 40 min duration under ambient conditions. Absorption activity was negatively correlated with increasing moisture content, and positively correlated with an increasing percentage of absorbent retained on the #8 sieve.								
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CONTENTS

INTRODUCTION	1
METHODSAbsorption Activity TimeSieve Tests	1
RESULTS CO ₂ Absorption Activity Sieve Tests Combined Influences	2
CONCLUSIONS	. 5
RECOMMENDATIONS	. 6
REFERENCES	. 8
APPENDIX A. MeshFit Computer Screen Printouts A.	1

ILLUSTRATIONS

Figure No.		Page No.
1	Rotap shaker with 5 sieves.	1
2	Effect of moisture content on activity time.	2
3	Influence of absorbent age on activity and moisture.	2
4	Variation in % retained on #8 mesh screen.	3
5	Fit of the multiple linear correlation.	3
6	Residuals for the multiple linear correlation.	3
7	Correlation between activity time and % retained.	4
	TABLES	
Table No.		Page No.
1	Output from S-Plus multiple linear regression of Sofnolime data.	5

GLOSSARY

ANU	Authorized for Navy Use List (NAVSEAINST 10560.2 series)
bar	Metric unit of pressure conveniently sized for supply pressures. One bar = 100 kPa, or 14.5 psi.
cmH₂O	A metric expression of static pressure head. One cmH $_2$ O = 0.01 meters of pure water. In pressure equivalents, 1 cmH $_2$ O = 0.7 torr, 98 Pa, or .098 kPa.
FIO ₂	Fraction inspired O ₂ . The fraction of the inspired gas composed of oxygen.
fsw	Feet of Seawater, a unit of pressure. One fsw = 0.3063 msw.
J/L	Joules per liter, unit of measure for "Work of Breathing" normalized for tidal volume. One $J/L = 1$ kPa.
kPa	Kilopascals or newton/m², unit of pressure. One kPa ~ 10.2 cmH₂O
msw	Meters of Sea Water. One msw = 3.2646 fsw.
NAVSEA	Naval Sea Systems Command
NEDU	Navy Experimental Diving Unit
psi	Pounds per Square Inch, an English measure of pressure. One psi = 6.9 kPa. 1 bar = 14.5 psi.
$\overline{ ext{P}}_{ ext{v}}$	Volume-averaged pressure, or resistive effort, otherwise known by the misnomer Work of Breathing (WOB). A computer derived estimate of total resistive respiratory effort obtained when breathing a UBA with a mechanical breathing simulator.
RMV	Respiratory Minute Volume with units of L \cdot min ⁻¹ . In scientific publications, this is referred to as expired ventilation (\dot{V}_E).
STPD	Standard temperature (0°C), pressure (1 atm abs), dry.
$\dot{V}O_2$	Metabolic oxygen consumption in L \cdot min ⁻¹ at STPD conditions.

INTRODUCTION

NEDU conducted quality assurance tests of non-indicating 408 Sofnolime for use in 30° F to 35° F (-1.1°C to 1.6°C) water. Three tests were run on each lot; sieve testing to determine the distribution of sodalime granule sizes within each sample, the NATO CO_2 absorption activity test, and moisture analysis. The following lot numbers were tested, 119082, 235076, 239076, 293086, 318092, 421116, 425116, 426116, 428116, 509017, 510017, 895115, 920115, 921115, 923115, 924115, 007102G and 738125G43.

METHODS

Absorption Activity Time

The CO₂ absorbent test bed was contained within a cylindrical glass tube¹ of 30 mm i.d., open on both ends, with a contained volume of 105 ml. The standard test setup was modified to include a custom designed and NEDU fabricated cooling jacket. A Neslab chiller (model RTE-111, Neslab Instruments Inc., Portsmouth, NH) circulated propylene glycol chilled to 28° F through the cooling jacket. The temperature within the vertically oriented test bed, 1.6 in. (4.1 cm) down from the top of the bed, was measured by a YSI 708 thermistor coupled with a thermistor thermometer (model 8502-12, Cole-Palmer Instrument Company, Vernon Hills, IL). In accordance with the operations manual¹ for the NEDU-NATO CO₂ absorbent test bench, a test gas of 50,000 ppm CO₂ in nitrogen was flowed through the test bed at a rate of 3.15 L·min⁻¹ (ATPD) at room temperature (~71°F). The gas flow was started,

and therefore the test begun, once the bed temperature registered 32°F or below.

Separate sodalime samples were used for moisture and mesh size analyses¹. A minimum of five samples from each lot were run through all three tests, with one operator performing all tests, except as described below.

Sieve Tests

Sieve tests were conducted on a Rotap shaker (Figure 1) with the entire 100 g sample from each of the 18 lots of Sofnolime 408 being placed initially on the top sieve. The U.S. mesh sizes for the sieves were 3.5, 5, 8, 10, 14, and 30, with a bottom pan which collected all the fine material passing through the #30 sieve. The averages of the grams

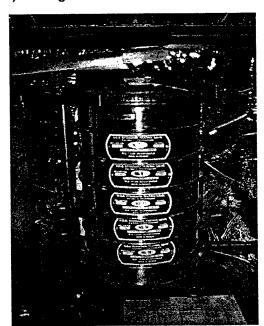


FIGURE 1. ROTAP SHAKER WITH 5 SIEVES IN PLACE.

(i.e., the %) retained on each sieve for each lot were entered into *MeshFit* software developed at NEDU. That software has within it a table of 135

Gaussian distributions of mesh size. Of those, only 46 met the existing British (and draft U.S. specification) for sodalime absorbent. The software determined which of those 46 distributions most closely matched the measured mesh size distribution, and then determined whether the measured distribution and the closest specification-meeting distribution (called the model distribution) were statistically different as determined by the Chi-square test. If the measured and model distributions were different, then the sodalime sample was outside of specification.

The software also returned the standard deviation of the model distribution. Screen prints from the MeshFit software are included at the end of this report.

The statistical analysis software S-Plus 4.0 by Mathsoft, Inc. was used to find correlations and regression coefficients for the following mean data on each sample: NATO tube test duration, % moisture, % absorbent retained on each sieve, mean sieve size, standard deviation of the model distribution, and a binary code for meeting specification (0 if met, 1 if not met).

RESULTS

CO₂ Absorption Activity

The average time to breakthrough in the NATO bench test was 7 min, far below the customary 40 min duration for 4-8 mesh sodalime when tested at room temperature. Under these cold conditions, an increase in absorbent

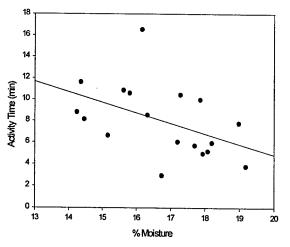
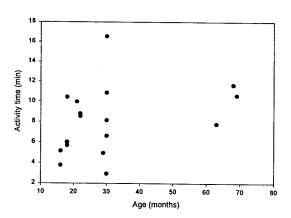


FIGURE 2. EFFECT OF MOISTURE CONTENT ON ABSORBENT ACTIVITY TIME.



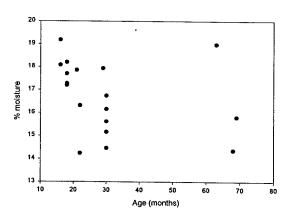


FIGURE 3. INFLUENCE OF ABSORBENT AGE ON ACTIVITY (TOP PANEL) AND MOISTURE (BOTTOM PANEL).

moisture was correlated with a reduction in absorbent activity, as shown in Figure 2. The mean moisture content was within the acceptable range² for all lots. Neither mean activity time nor percent moisture content was related to the age of the absorbent (Figure 3).

In addition to the 90 sample runs, two lots were tested by different operators to gain information on operator based variability. The mean activity times and moisture content did not differ significantly between the operators.

8 mesh Sofnolime 408

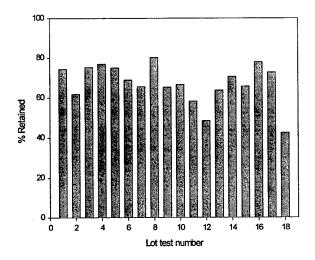
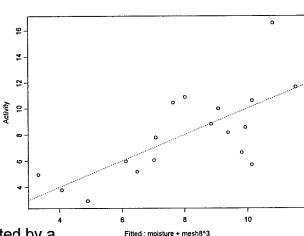


FIGURE 4. VARIATION IN % RETAINED ON #8 MESH SCREEN WITH LOT NUMBER.

Sieve Tests

The amount of absorbent retained on the #8 sieve was surprisingly variable (Figure 4), ranging from 43% to 80% of the total 100 g sample (67% ± 10%, mean ± SD). Three of the 18 samples were outside of the British specification for Sofnolime, and are indicated on the screen prints by the following text in red: "Significantly different from best model!".



Combined Influences

NATO tube test results could be predicted by a multiple linear correlation (Table 1, Figures 5 and 6) combining the positive effects of the percentage of 8 mesh material and the negative effects of % ...

A plot of the residuals for the fit (Figure 6) aid in both assessing the adequacy of the fit, and in identifying outliers³, such as the upper right data point in Figure 6. In this case, deleting the identified outliers from analysis did not appreciably change the results of the regression.

FIGURE 5. FIT OF THE MULTIPLE LINEAR CORRELATION.

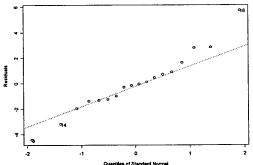


FIGURE 6. RESIDUALS FOR THE MULTIPLE LINEAR CORRELATION.

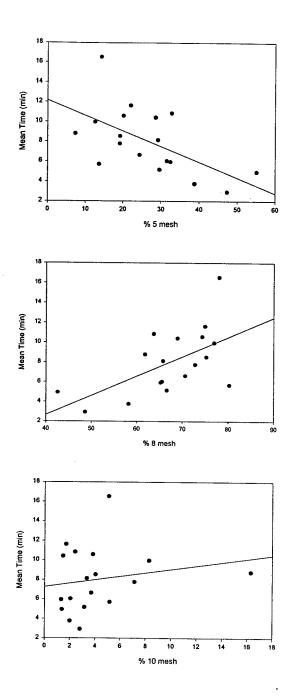


FIGURE 7. CORRELATION BETWEEN ACTIVITY TIME AND % RETAINED ON THE NUMBER 5, 8, AND 10 SIEVES.

Due to the effects of colinearity (the percentage retained on one sieve was strongly dependent on the amount retained on the other sieves; Figure 7), the information on the other mesh percentages did not improve the fit to the data.

There is a theoretical advantage to narrow granule size distributions. However, the standard deviations of the model distributions varied only slightly (range 0.7 to 0.9) and thus exhibited no noticeable effect on activity times in the NATO tube test. However, the effect of the percentage of material trapped on the #8 sieve was strong. The best correlation was as follows:

Minutes Activity = $17.39 - (0.85 \cdot \text{%moisture}) + (0.000015 \cdot \text{% retained on } #8 \text{ sieve})$

Below is the output from the multiple linear regression. The F statistic for the fit was significant (P = 0.005).

```
*** Linear Model ***

Call: lm(formula = Activity ~ moisture + mesh8^3, data = SOFcold, na.action = na.omit)

Residuals:

Min 1Q Median 3Q Max
-4.439 -1.303 -0.1143 0.835 5.738

Coefficients:

Value Std. Error t value Pr(>|t|)

(Intercept) 17.3922 6.9255 2.5113 0.0240

moisture -0.8493 0.3899 -2.1780 0.0458

I(mesh8^3) 1.5 10^-5 0.5 10^-5 2.9883 0.0092

Residual standard error: 2.489 on 15 degrees of freedom

Multiple R-Squared: 0.5056

F-statistic: 7.669 on 2 and 15 degrees of freedom, the p-value is 0.005078
```

TABLE 1. OUTPUT FROM S-PLUS MULTIPLE LINEAR REGRESSION OF SOFNOLIME DATA. ACTIVITY = ACTIVITY TIME (MIN), MOISTURE = % MOISTURE, MESH8 = % OF ABSORBENT SAMPLE RETAINED ON #8 MESH SIEVE.

CONCLUSIONS

This work was motivated by the field observation that in cold water some CO₂ absorbent canisters were obtaining much shorter durations than other canisters. To help explain this apparent anomaly, NEDU was asked by the Fleet to study the physical and chemical absorption characteristics of specific lots of 408 Sofnolime.

When the NATO test bench was chilled by a temperature controlled water jacket, activity time was markedly decreased from a usual duration of 40 min to an average of 7 min. The activity times for various lots became highly variable, on a percentage basis.

The activity time of the NATO tube tests run under cold water conditions correlated with both absorbent moisture content and mesh size. For 408

Sofnolime, the average mesh size should be about 6. When examining lot-to-lot variability, if more material collects on the #8 sieve on one lot than another, then that lot has a greater percentage of small granules than the other lot. An increase in the amount of small granules is associated with improved performance, e.g. longer duration, on the NATO tube test.

When NATO tests are run under room conditions, the critical dependence of absorption activity on mesh distribution is not apparent. The differences between lots are accentuated under cold conditions, leading to finer grain absorbents being preferred over larger grain absorbents. This leads to the suggestion that for cold water use, 812 Sofnolime may be preferable to 408 Sofnolime if the attendant increase in breathing resistance can be tolerated.

We currently do not fully understand the observed interaction between granule size distributions and cold water. However, two soon to be released NEDU reports will shed some light on granule size distributions and the trade-off between sodalime absorption performance and breathing resistance^{4,5}. A third report is being drafted which develops discrete models of CO₂ accumulation rates in single granules incorporating the effects of thermal flux between "shells" within the granule, heats of reaction, temperature dependent diffusion rates, and diffusion barriers formed by reaction products (water and carbonate) within the granule.

The next step in understanding the current observations is to expand the existing kinetic model to include overall performance of a bed of absorbent with varying water content and granule size distributions.

RECOMMENDATIONS

The NATO bench test results should not be used to predict UBA canister durations. However, we do believe that there is a qualitative relationship between NATO tube test results and UBA performance. Material identified as being of poor quality in the NATO quality assurance tests is likely to perform poorly in an actual UBA canister.

Consequently, we make the following recommendations: to maximize absorbent performance and consistency in cold water, sodalime lots with a large percentage of material retained on the #5 sieve should not be used for cold water applications. Moisture content should be monitored, and buckets with moisture contents of 18% or above should also not be used for cold water applications.

There are at least three ways to implement these recommendations, ranked in order of increasing difficulty for the user:

1) Require lot specific reports of moisture content and sieve distribution from the

manufacturer. Unfortunately, this information is not available when ordering from standard stock. The manufacturer's test results may not apply if the material is repackaged or is stored for a long period of time before use, unless sodalime buckets are hermetically sealed.

- 2) Send samples from each lot to NEDU or other testing laboratory for analysis.
- 3) Have the users perform lot testing. This would require at a minimum a drying and weighing apparatus, a ROTAP sieve (Fig. 1), and training.

Alternatives to these recommendations would be to accept the lot variability in the cold, or keep the absorbent warm. The suggestion that fine grain (10-14 U.S. mesh, 8-12 British mesh) absorbent should perform better in the cold will be tested at NEDU in the near future. Unfortunately, fine grain material is more expensive than large grain (4-8 mesh) absorbent, and imposes a greater breathing resistance.

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- 3. *Biometry*, R.R. Sokal, F.J. Rohlf., 3rd edition, W.H. Freeman and Co., NY, pg. 531.
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- 5. J.R. Clarke. Size Distributions for Sodalime Absorbent Granules What Do They Tell Us?, Draft NEDU report.

APPENDIX A. Meshfit Computer Screen Printouts

